FIB-TEM INVESTIGATIONS INTO THE CONDITION OF REFRACTORY PRESOLAR PHASES AFTER STARDUST-LIKE IMPACTS. T. K. Croat[1], C. Floss[1], S. Sosothikul[1], F.J. Stadermann[1], A. T. Kearsley[2], and M.J. Burchell[3] [1]Laboratory for Space Sciences and Department of Physics, Washington University, St. Louis, MO 63130, USA, tkc@wustl.edu [2]Department of Mineralogy, Natural History Museum, London SW7 5BD, United Kingdom, [3] Centre for Astrophysics and Planetary Science, School of Physical Sciences, University of Kent, Canterbury CT2 7NH, United Kingdom.

Introduction: The presolar grain abundance in Wild2, as determined from NanoSIMS measurements of Al foil craters, was considerably lower than anticipated [1]. However, there were large uncertainties in this determination due to the difficulties associated with SIMS measurements on cratered foils and due to possible grain destruction or modification on impact. More recent NanoSIMS results from Acfer 094 test shots show a ~15x reduction in the presolar grain abundance in Al foil craters below that seen in the matrix material used for the shots [2, 3]. This and other results [4] suggest significantly higher Wild 2 presolar grain abundances, although with large uncertainties. Here, we present FIB-TEM results from STARDUST-analogue test shots made with a mixture of refractory minerals (including SiC), with the goal of better understanding the conditions and survivability of common presolar phases under STARDUST-like impact with Al foils.

Samples and Experimental Methods: A mixture of refractory materials (alumina, SiC, Si_3N_4, TiC, TiN, diamond and olivine glued together with WN acrylate) was shot at Al 1100 foil with the two stage light gas gun at the University of Kent [5] with an impact speed of 6.05 km/s. The resulting foil craters were then examined with SEM-EDXS and Auger spectroscopy to identify and characterize surviving projectile material [6]. FIB sections were taken from four craters (with diameters from 2.0 to 6.5 microns) that showed evidence of Si-rich and Ti-rich particles (e.g. Figs. 2 and 3 from [6]), and the crystal structures and chemical compositions of the surviving material were determined with TEM.

Results: SEM-EDX spectra of craters can identify the predominant impactor type (e.g. Ti-rich or Si-rich) and can approximately determine the spatial distribution of the surviving material within the crater [6], although there are some problems with signal strength based on the crater topology. However, all spectra are dominated by Al, and any C and N peaks are absent or very weak.

Ti-rich craters: FIB-TEM sections were made from two Ti-rich craters with 5-6 μm diameters, and cross-sectional views reveal their shape and depth (e.g. Fig. 1a). In the first Ti-rich crater, five discrete Ti-rich grains with diameters from 115 to 420 nm were found along the crater bottom (shaded blue in Fig. 1b). Diffraction data taken from three grains were indexed to a 4.2 ± 0.1 Å FCC phase, based on {011} or {001} patterns (e.g. Fig. 1c), and are consistent with both TiC (FCC; a=4.35Å) and TiN (FCC; a=4.24Å). TEM-EDX spectra (in particular the N/C count ratios) can be used to differentiate between surviving TiN and TiC particles. TEM-EDX of unfired TiN and TiC particles showed significantly different N/C count ratios (of 1.5 ± 0.3 and 0.50 ± 0.15 for TiN and TiC, respectively). The Ti-rich grains in Ti crater 1 had N/C count ratios of 2.7 ± 0.8, and thus are clearly TiN. In a second Ti-rich crater, several surviving Ti-rich grains were also found. Although their crystal structures have not yet been determined, these Ti-rich grains had lower N/C ratios (0.45 ± 0.06) that are consistent with TiC.

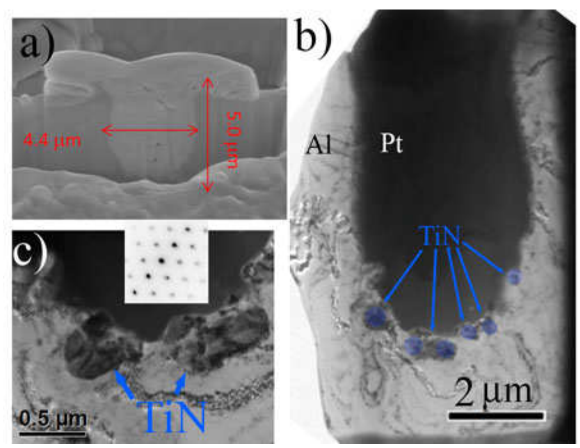

Fig.1. a) FIB-SE image of Ti crater 1 during thinning process showing Al crater outline (crater filled and topped with brighter Pt) b)TEM image of thinned FIB section showing five Ti-rich grains (shaded in blue) at bottom c) TEM image of several TiN grains at crater bottom with inset showing a [011] FCC diffraction pattern.

Si-rich craters: Like the Ti-rich craters described above, the larger 5 μm diameter Si-rich crater contained several surviving Si-rich grains (with 200 and 250 nm diameters) found near the crater bottom (Fig. 2a). One grain was identified as 3C-SiC based on diffraction patterns from [112] and [101] FCC zones, and

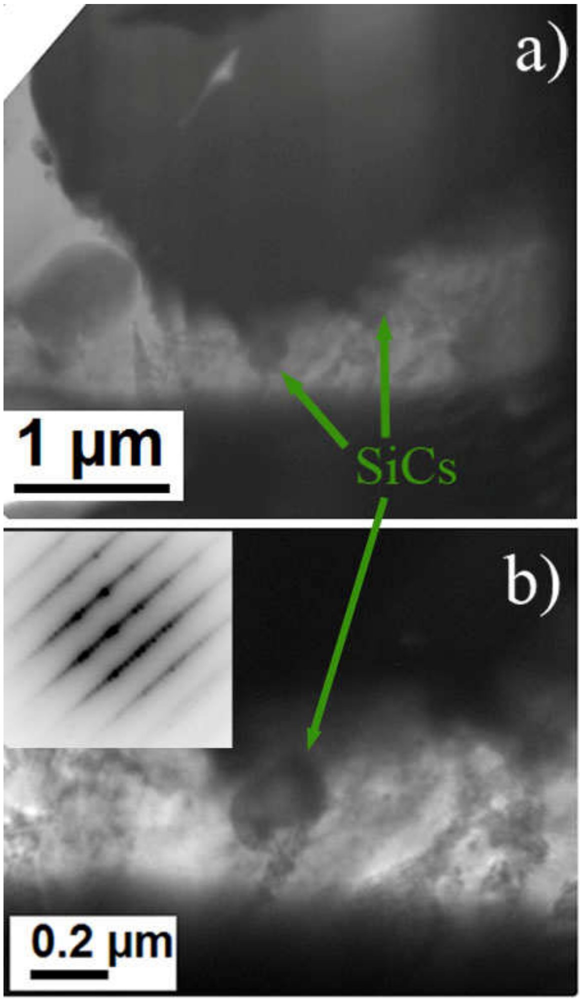

Fig. 2. a) TEM image of larger Si-rich crater with two SiCs found at the crater bottom and b) image and diffraction pattern of surviving SiC at [101] orientation.

stacking faults/twins were apparent (Fig. 2b). EDX spectra showed N/C count ratios of 0.35 ± 0.07 and were consistent with known SiCs. Since SiC grains were not identified in the unfired projectile material [6], comparison between surviving SiCs and unfired ones was not possible in this case.

In a second smaller Si-rich crater (~1.4 μm diameter; Fig. 3a), a single surviving silicon nitride grain with dimensions of 570 x 480 nm was found directly at the crater bottom (Fig 3b). The phase was identified as Si_3N_4 (hex; a= 7.8Å, c=5.6 Å) based on diffraction patterns collected from 7 major zone axes. Fig. 3b shows the grain along a high-symmetry orientation with the crystal faces clearly visible, suggesting that the grain did not melt and resolidify on impact. TEM-EDX spectra show N/C count ratios of 2.00 ± 0.01, slightly higher than N/C ratios in unfired Si_3N_4 grains, but still consistent with identification as a nitride phase.

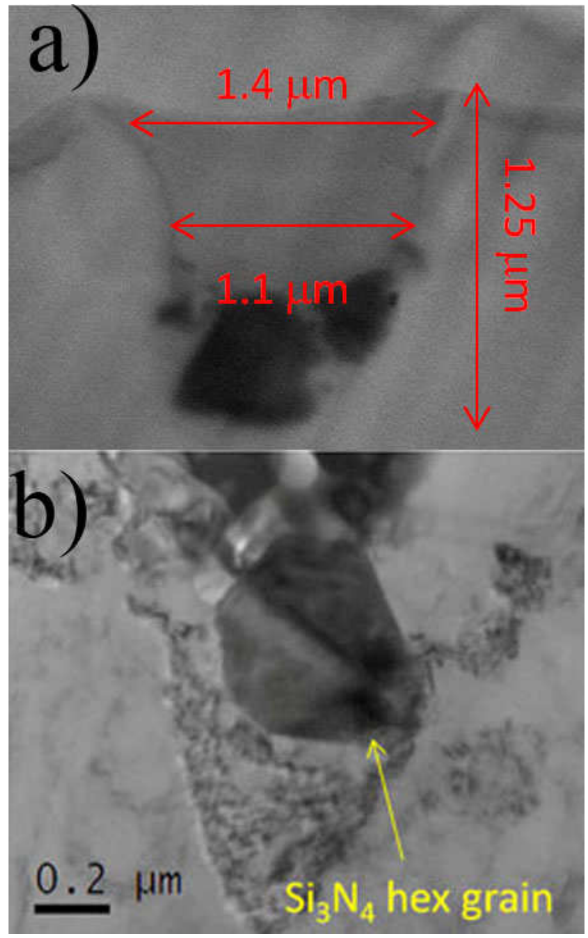

Fig. 3 a) FIB-SEM image of smaller Si-rich crater cross-section and b) TEM image of large surviving Si_3N_4 grain at its crater bottom.

Discussion: The above results demonstrate that sub-micron grains of common refractory presolar phases (such as SiC) can survive STARDUST-like impacts, and that significant grain destruction or loss on impact does not always occur. Even for the least refractory of the four surviving phases reported here (which is Si_3N_4), the surviving grain (Fig. 3) is roughly the same size as the typical incident projectiles (which had average diameters of ~700 nm; [6]), suggesting that little material was lost on impact. Comparable FIB-TEM studies of silicate and oxide test shot samples are planned in an attempt to determine the cause of the significantly reduced detection [2, 3] of such presolar grains.

References: [1] Stadermann F.J. et al. (2008) *MAPS 43*, 299. [2] Floss C. et al. (2013) *ApJ*, in press. [3] Floss C. et al. (2013) *LPS XLIV, Abstract # 1133*. [4] Leitner J. et al (2012) *LPS XLIII, Abstract # 1839*. [5] Burchell M.J. et al. (1999) *Measure. Sci. Tech, 10*, 41. [6] Croat et al. (2011) *LPS XLII, Abstract # 2520.*